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IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :  
Yoshihiro KURII, et al. : EXAMINER: Umez-Eronini, Lynette T.  
SERIAL NO. 09/912,318 :  
FILED: July 26, 2001 : GROUP ART UNIT: 1765  
FOR: MICRO-ETCHING COMPOSITION FOR COPPER OR COPPER ALLOY,  
MICRO-ETCHING METHOD, AND METHOD FOR MANUFACTURING PRINTED  
CIRCUIT BOARD

DECLARATION UNDER 37 CFR 1.132

Sir:

Now comes **Sachiko NAKAMURA** who deposes and states:

1. That I am a graduate of Nara Women's University and received my bachelor's degree in the year of 1984.
2. That I have been employed by MEC CO., LTD. For 15 years as a chemical researcher in the field of printed circuit fabrication.
3. I am a co-inventor of the above-identified application.
4. In order to demonstrate the surprising nature and advantages of the present invention over the reference Ferrier et al. (USP 5,869,130), the following Comparative Experiment was conducted by me or under my supervision:

Comparative Example 6

Micro-etching agents (Composition 1 and Composition 2) were prepared by mixing the components shown in the Table 2. Composition 1 is the same micro-etching agent as used in Example 5 of the present invention. Composition 2 is the same micro-etching agent as used in Example 6 of the present invention, in which cresol sulfonic acid that is used in Composition 1 is omitted.

#### <Evaluation of solder heat resistance>

Copper was dissolved in Composition 1 and Composition 2 to make a copper concentration of 50 g/l, which is a high concentration occurring in continuous use of micro-etching agents.

The Compositions 1 and 2 were sprayed onto glass fabric epoxy resin-impregnated copper-clad laminated boards (FR-4 grade), the both sides of which were clad with a copper foil with a thickness of 18  $\mu\text{m}$  at 25°C, thereby etching 2 $\mu\text{m}$  of the copper surface.

The glass fabric epoxy resin-impregnated prepreg (FR-4 grade) was laminated onto each side of the resulting copper-clad laminated boards and pressed. The periphery was cut off to prepare test pieces. A 2 atmospheric pressure load was applied to the test pieces for 4 hours at 121°C and 100% RH using a pressure cooker. The test pieces were immersed in a molten solder bath at 270°C for 60 seconds according to JIS C6481 and removed from the bath to evaluate swelling (upholding from the surface) of the prepreg solder by visual observation. The results are shown in Table 2.

Samples of test pieces 1 and 2 are attached hereto. The copper surface treated with a micro-etching agent containing cresol sulfonic acid differs from that of the copper surface treated with a micro-etching agent not containing cresol sulfonic acid after etching. For this reason, the test piece 2 is darker than the test piece 1.

#### <Peel-off strength>

Copper was dissolved in Composition 1 and Composition 2 to

make a copper concentration of 50 g/l.

The micro-etching compositions were sprayed onto the shiny side of copper foils with a thickness of 70  $\mu\text{m}$  at 25°C, thereby etching 2  $\mu\text{m}$  of the copper surface.

The above prepreg was laminated and pressed onto the treated surface of the copper foil. To measure the peel-off strength, the copper foil was removed except for the area of a stripe with a width of 1 cm according to JIS C6481. The results are shown in Table 2.

TABLE 2

	Amount (g/l)		Micro-etching agent before dissolving copper		Micro-etching agent after dissolving copper (50 g/l)	
			Solder heat resistance	Peel-off strength (N/mm)	Solder heat resistance	Peel-off strength (N/mm)
Comp. 1	Sulfuric acid	200	No prepreg swelling	1.3	No prepreg swelling (Test piece 1)	1.1
	Hydrogen peroxide	50				
	5-Phenyltetrazole	0.35				
	Cresol sulfonic acid	10				
	Sodium chloride (Chloride ion conc.)	10 ppm				
	Ion exchanged water	(Balance)				
Comp. 2	Sulfuric acid	200	No prepreg swelling	1.3	Prepreg swelled (Test piece 2)	1.0
	Hydrogen peroxide	50				
	5-Phenyltetrazole	0.35				
	Sodium chloride (Chloride ion conc.)	10 ppm				
	Ion exchanged water	(Balance)				

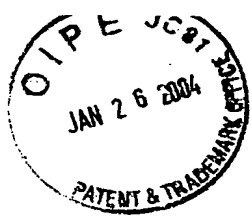
As can be seen from Table 2, cresol sulfonic acid is very effective for improving the solder heat resistance when a large amount of copper is dissolved. No cited references describe an increase in the solder heat resistance due to the addition of cresol sulfonic acid.

5. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

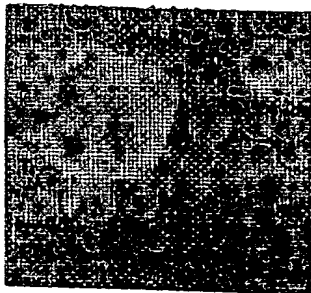
6. Further deponent saith not.

Sachiko Nakamura  
Sachiko NAKAMURA

Jan. 9, 2004  
Date



Test Piece 1:



Test Piece 2:

